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Effect of Sterilization Irradiation on Friction and Wear of Ultrahigh- Molecular-Weight Polyethylene

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SUMMARY

The effect of sterilization gamma irradiation on the friction and wear properties of ultrahigh-molecular-weight polyethylene (UHMWPE) sliding against 316L stainless steel in dry air at 23⁰ C was determined. A sliding pin-on-disk apparatus was used. Experimental conditions, in most cases, included a 1-kilogram (9.8-N) load, a 0.061- to 0.27-meter-per-second sliding velocity, and a 32 000- to 578 000-meter sliding distance.

Although sterilization doses of 2.5 and 5.0 megarads greatly altered the average molecular weight and the molecular weight distribution of UHMWPE, the friction and wear properties of the polymer were not significantly changed. Average wear rates of 1.6, 1.3, and 1.3×10^{-15} cubic meter per newton-meter were obtained for the unirradiated and the 2.5- and 5.0-megarad-irradiated specimens, respectively. The corresponding average friction coefficients for these three irradiation levels were 0.44, 0.44, and 0.38, respectively.

INTRODUCTION

The surgical replacement of diseased or damaged human joints with artificial devices has become commonplace. During 1976, there were 80 000 total-hip replacements and 30 000 knee replacements and in the United States (ref. 1). Most presently used prostheses consist of either a cobalt-chromium-molybdenum alloy (Vitallium) or surgical stainless steel (316L) articulating against ultrahigh-molecular-weight polyethylene (UHMWPE).

The conventional sterilization technique of steam autoclaving cannot be used with polyethylene components because of their tendency to deform under those conditions. Normally, polyethylene must be sterilized by gamma irradiation (2.5-megarad dose). Gamma irradiation is known to cause crosslinking in polyethylene (ref. 2). Crosslinking, in turn, alters polymer molecular weight, crystallinity, elastic modulus, and other physical properties.

Rostoker and Galante (ref. 3) show that changes in molecular weight (MW) due to variations in molding temperature can affect the wear properties of UHMWPE. Miller, et al. (ref. 4) also report lower wear rates with molded (higher MW) than with machined (lower MW) UHMWPE. Similar results were obtained by Seedhom, et al. (ref. 5).

Matsubara and Watanabe (ref. 6) studied the effect of gamma irradiation on the friction and wear properties of high-density polyethylene. In general, wear increased

with increasing radiation dose. Awatani and Minegaki (ref. 7) show a wide variation in the wear properties of gamma-irradiated polyethylene depending on the radiation atmosphere (air or vacuum). Recently, Shen and Dumbleton (ref. 8) studied the effect of high gamma doses (20 to 1000 megarads) on the friction and wear properties of UHMWPE. In general, both friction and wear increased with increasing dose. It was concluded that these high gamma doses would not produce a useful bearing material. However, there have been few studies of radiation effects at low doses (2.5 megarads), corresponding to sterilization conditions. Ungethym (ref. 9) reports some limited data on the effect of low radiation doses (0 to 15 megarads) on polyethylene wear in pin-on-disk tests. Wear increased with increasing dose, but no information is provided as to the test conditions other than load (68 and 98 N).

Therefore, the objective of this investigation was to determine the effect of sterilization irradiation on the friction and wear properties of UHMWPE sliding against 316L stainless steel in dry air at 23° C. A pin-on-disk apparatus was used. Experimental conditions, in most cases, included a 1-kilogram load, a 0.061- to 0.077-meter-per-second sliding velocity, and a 32 000- to 578 000-meter sliding distance. Three irradiation dose levels were studied: 0, 2.5, and 5.0 megarads.

Mr. Casper Stark of Howmedica, Inc., irradiated the polymer specimens, and Mr. Gene Farling of Zimmer-USA provided the disk specimens.

APPARATUS

The pin-on-disk sliding friction apparatus is shown in figure 1. The test specimens were mounted inside a plastic chamber. This allowed the moisture content of the test atmosphere to be controlled. A stationary 0.476-centimeter-radius hemispherically tipped polymer rider was placed in sliding contact with a rotating 6.3-centimeter-diameter (1.22-cm-thick) metal disk. Constant sliding speed in the range 0.061 to 0.27 meter per second was maintained. A normal load of 1 kilogram was applied with a deadweight.

MATERIALS

Rider specimens were made of ultrahigh-molecular-weight polyethylene. The riders were machined from a block of surgical-grade polymer (RCH1000) manufactured and supplied by Hoechst A. G. After machining, the riders were gamma irradiated in air at atmospheric pressure to doses of 2.5 and 5.0 megarads. A cobalt-60 source was used.

The polished metal disks were made from surgical-grade stainless steel 316L, supplied by Zimmer-USA, that had a Rockwell B hardness of 75 to 77. After original fabrication, the disks were rough ground with 180-grit silicon carbide paper and a water coolant. The specimens were then successively ground with finer grit papers of the same type until a 600-grit finish was obtained. A polishing operation followed in which 6-micrometer diamond paste on a nylon cloth was used with kerosene as the lubricant and coolant. The final polishing was done with a short-nap microcloth of 0.05-micrometer gamma alumina with distilled water as the lubricant and coolant. Final surface roughness was about 0.038 micrometer centerline average (CLA) (1.5 μ in.), which is comparable to that of commercial prosthesis components.

TEST PROCEDURE

Disk and rider specimens were scrubbed with a commercial (nonabrasive) detergent, rinsed thoroughly with distilled water, and then dried on clean filter paper. The specimens were assembled in the test chamber. The chamber was purged with the test atmosphere, dry air (<50-ppm H₂O), for a minimum of 10 minutes. The disk was set in motion and the rider loaded against it. Frictional force was measured by a strain-gage transducer. Rider wear was determined periodically by stopping the test and measuring the wear-scar diameter. No attempts were made to separate creep effects from wear.

RESULTS

Wear

Effect of irradiation. - Table I summarizes the wear data and conditions for each test. Ten tests were performed with the unirradiated polymer, five with the 2.5-megarad-irradiated polymer, and seven with the 5.0-megarad-irradiated polymer. All tests except test 7 (0 megarad) were run with a 1-kilogram (9.8-N) load. Most tests were performed at 25 rpm, which yielded sliding velocities from 0.061 to 0.077 meter per second, depending on the disk wear-track circumference. Five tests, tests 8 to 10 (0 megarad) and tests 6 and 7 (5 megarads), were run at 100 rpm (0.24 to 0.27 m/sec). Tests are numbered sequentially at each irradiation level according to sliding distance, except for the high-load and high-speed tests.

Table II further summarizes the friction and wear results for the three irradiation levels. Both the 2.5- and 5.0-megarad levels yielded the same average wear rate, 1.3×10^{-5} cubic meter per newton-meter. This is about 20 percent less than the value

obtained for the unirradiated polymer, 1.6×10^{-15} cubic meter per newton-meter. However, statistical analysis of these data using Student's *t* distribution indicates that this difference in wear rate is not significant at a 95-percent confidence level. Therefore, we can only conclude that sterilization irradiation to 5.0 megarads does not significantly change the wear properties of the polymer.

Effect of load. - This investigation was not intended to be a complete parametric study. Therefore, only a few tests were performed at conditions other than the standard ones previously stated. One test, test 7 (0 megarad), was run at a 2-kilogram (19.6-N) load rather than the standard 1-kilogram (9.8-N) load. This test yielded a wear rate (in $\text{m}^3/\text{N-m}$) that was within the data scatter of the remaining unirradiated-specimen tests.

Effect of sliding velocity. - Five tests, tests 8 to 10 (0 megarad) and tests 6 to 7 (5 megarads), were performed at 100 rpm (0.24 to 0.27 m/sec) rather than the standard 25 rpm (0.061 to 0.077 m/sec). Comparing the wear rates for these five tests with the lower speed results does not reveal any speed effect.

Effect of contact stress. - Because of the test geometry, the contact stress obviously decreased as the test progressed and the rider wear-scar area increased. Contact stress is shown as a function of wear-scar diameter for a constant 1-kilogram load in figure 2. The stress is about 13 megapascals (1830 psi) for a 1-millimeter wear-scar diameter. This is the wear-scar diameter that is normally observed after the first few hundred meters of sliding. Then the contact stress rapidly decreased as the wear-scar diameter increased, until it reached 0.79 megapascal (110 psi) at a wear-scar diameter of 4 millimeters. This is the wear-scar diameter usually observed after several hundred thousand meters of sliding at test conclusion.

Wear volume as a function of sliding distance (or decreasing contact stress) is shown in figure 3 for the unirradiated and the 2.5- and 5.0-megarad-irradiated specimens, respectively. In general, these log-log plots do not show any large changes in wear rate. However, if these data are plotted in the form of incremental wear rates ($\text{m}^3/\text{N-m}$) (i. e., wear rates between each wear-scar measurement) as a function of sliding distance, differences are more obvious. These data are shown in figure 4. The run-in effect is quite obvious below 3000 meters. The incremental wear rates for the unirradiated polymer (fig. 4(a)) appear to level out after run-in. Only test 10 yielded some late wear-rate increases. There appears to be a trend toward lower wear rates for the 2.5-megarad-irradiated polymers (fig. 4(b)) at longer sliding distances (or lower contact stresses). This trend is more obvious for the 5.0-megarad-irradiated specimens (fig. 4(c)).

Friction

Effect of irradiation. - Average friction coefficients for each test are given in table I. Average friction values for each irradiation level are given in table II. The 5.0-megarad-irradiated polymers had an average friction coefficient of 0.38, compared with 0.44 for both the unirradiated and 2.5-megarad-irradiated specimens. This 15-percent difference is probably not significant when the standard deviations are considered.

Effect of sliding distance. - During run-in the friction coefficients gradually increased for all tests until a fairly steady value was reached after 2000 to 3000 meters of sliding. For the unirradiated specimens, the friction reached a maximum and then declined during the latter part of the test (sliding distances less than 20 000 m) as shown in figure 5. For the 5.0-megarad-irradiated specimens, the friction coefficient was essentially level after run-in; this behavior is illustrated in figure 6. The 2.5-megarad-irradiated specimens yielded both types of behavior.

Polymer Characterization

The polymer riders from test 3 (0 megarad), test 3 (2.5 megarads), and test 5 (5 megarads) were studied by solution-viscosity techniques (ref. 10) as well as by differential scanning calorimetry. These data are given in table III, along with microhardness data for these specimens. The characterization parameters are fully described in the appendix. The riders were cut into four quadrants, the immediate surface layer was discarded, and samples were shaved from the central-axis area of the quadrants. Each test was repeated until results were within ± 2 percent, except for the hardness measurements.

Solution-viscosity measurements. - As shown in table III, gamma irradiation caused a rather drastic change in the average molecular weight and in the molecular weight distribution. The amount of insoluble gel (i.e., $MW > 6 \times 10^6$) was increased four times for the 2.5-megarad-irradiated specimens and over five times for the 5.0-megarad-irradiated specimens. The irradiation also decreased the weight-average molecular weight of the soluble polymer fraction by over an order of magnitude. In contrast, the amount of very low-molecular-weight polymer ($< 20\,000$) was increased by irradiation. Obviously, there are competing processes of chain scission and chain crosslinking.

Crystallinity measurements. - There was a slight increase in crystallinity with increasing irradiation, whether it was determined by density changes or from differential scanning calorimetry (DSC). Values ranged from 68 to 72 percent from the density data and 46 to 50 percent from DSC.

Differential scanning calorimetry. - The DSC trace for the unirradiated polymer is shown in figure 7. It exhibits a single melting peak and melting temperature range typical of UHMWPE. Unusual melting behavior was observed for both irradiated specimens. This behavior is illustrated for the 2.5-megarad-irradiated specimens in figure 8. The melting range is quite similar to that for the unirradiated specimen, but now a double endotherm is present.

Hardness measurements. - The hardness measurements in table III are averages for several specimens. There appears to be no difference in hardness for the three irradiation levels.

DISCUSSION

Wear

Several investigators (refs. 11 to 13) have reported wear data for UHMWPE sliding against stainless steel under dry conditions. Tanaka and Uchiyama (ref. 11) measured the wear of superhigh-molecular-weight polyethylene with a pin-on-disk apparatus. A calculated wear rate of about 5×10^{-16} cubic meter per newton-meter was obtained at a load of 1 kilogram and a sliding speed of 0.10 meter per second. Dowson, et al. (ref. 12) report a lower wear rate (1.3×10^{-16} m³/N-m) with a tri-pin-on-disk apparatus. Data from the author's laboratory (ref. 14) yielded a much higher wear rate (1.5×10^{-14} m³/N-m) for UHMWPE articulating against Vitallium in a total-hip simulator. This value is comparable to the unirradiated-specimen value from table II (1.6×10^{-15} m³/N-m). The data from this paper are several times greater than the literature results but an order of magnitude less than those for the total-hip simulator (ref. 14). The abnormally high wear rate from the simulator is no doubt related to the short duration of the test (8 hr). This time represents only 110 meters of sliding and therefore reflects run-in effects. There were also obvious geometry differences between the total-hip simulator and the pin-on-disk apparatus.

The Tanaka work was performed on polyethylene from a source other than the one used in this study. This may explain the wear-rate differences. Dowson, however, used the same type of polyethylene (RCH1000), but there were differences in his test apparatus. He used truncated cones for the polymer specimens rather than hemispherical riders. The lower initial contact stresses obtained with the cones should yield lower run-in wear. In addition, he ran the three test riders on the same track, and this would probably affect the transfer-film characteristics. The difference in wear rate might be related to these factors.

Another reason for higher wear rates is that polymer creep can cause apparent increases in the wear scars that are not related to the wear process. However, stati-

cally loaded (19.6 N) riders with preformed wear scars showed no measurable change in scar diameter in tests as long as 2 weeks. Although other investigators (ref. 15) have shown creep to be important in wear measurements, it did not appear to be a major factor in this study.

Irradiation Effects

It is well known that thin, oriented polymer transfer films on a metal substrate can greatly affect friction and wear properties (refs. 16 to 20). It has been shown that a smooth molecular profile enhances this process and that bulky side groups or radiation-induced crosslinking hinders it. Indeed, Shen and Dumbleton (ref. 8) did not detect transfer films at all with irradiated polyethylene (20 to 1000 megarads). Because of the radiation-induced changes (table III) in the polymers of this study, we might expect differences in transfer-film characteristics. However, optical microscopy of these films did not show any obvious differences. The transfer films formed readily during run-in and did not appear to change during the remainder of the test. Transfer consists of small (<1 to >5 μm diam) discrete particles to much larger (10- to 70- μm major dimension) patches. However, patches were only occasionally observed. Examples of these types of transfer film are shown in figure 9. The thickness of the large patch can be estimated from interference colors to be between 0.4 and 0.8 micrometer. The small transferred particles are 0.4 micrometer thick or less. The surface roughness on the disk wear track attained a steady level of about 0.10 micrometer CLA (4 $\mu\text{in.}$) after run-in.

Wear-Time Behavior

Some investigators (refs. 3, 12, and 21) report wear-rate increases with UHMWPE during the latter stages of some wear tests. Rostoker and Galante (ref. 3) report a late wear-rate increase (after 10^6 m of sliding) with graphite-filled UHMWPE. Dowson (refs. 12 and 21) also observed wear-rate increases with UHMWPE. These increases were attributed to a fatigue mechanism. The onset of this higher wear-rate regime began after 100 000 to 500 000 meters of sliding. Others (refs. 22) observed phenomena that might be fatigue related. However, these late wear-rate increases were generally not observed in the longer term (100 000 m) test of this study. Although one or two tests yielded some incremental wear-rate increases (such as test 10) most yielded fairly constant wear rates (after run-in) or a gradually decreasing rate. The latter effect was particularly noticeable with the 5.0-megarad-irradiated specimens.

PV Effects

It is known (ref. 23) that polymer wear rates are accelerated when sliding conditions reach a certain severity. The severity is measured by the product of contact stress and sliding speed (PV). Shen and Dumbleton (ref. 8) report a limiting PV for unirradiated UHMWPE of about 2×10^5 (Pa) (m/sec). They also found that this value was increased by gamma irradiation to 3.4×10^5 (Pa) (m/sec) for a 20-megarad dose. In the present study, test conditions were such that most tests would have continued above the limiting PV (i. e., 2×10^5 (Pa) (m/sec)). However, at the latter stages of some long-term tests, the contact stress can decrease to such a level that the limiting PV is reached. This would show up as a decrease in wear rate as a function of time, as described in the previous section. This effect should be more pronounced at the 5.0-megarad level because of the increasing PV limit, as observed.

Differential Scanning Calorimetry

As stated previously, the gamma-irradiated specimens exhibited unusual melting behavior, as illustrated in figures 7 and 8. The melting behavior of the unirradiated polymer (fig. 7) is typical of UHMWPE. However, the double endotherm present in the DSC trace for the 2.5-megarad-irradiated specimen (fig. 8) is unusual. This type of behavior is rarely seen with UHMWPE but is quite common in the fiber industry (refs. 24 to 26), where it has been linked to the presence of extended-chain crystals that are produced during fiber orientation. Double endotherms are due to two types of crystals each exhibiting its own melting behavior. Therefore, the irradiated-UHMWPE data may be related to extended chain formation or, more likely, to some radiation-induced effect. At any rate, when a sample of the irradiated polymer was melted, recrystallized, and remelted, the double peak was absent. Nor was there a double peak in the DSC trace for material taken from the rider surface. Since these riders were machined, it is possible that the frictional heat from the machining process caused the polymer to recrystallize in the normal mode.

SUMMARY OF RESULTS

The results from the study of the effect of sterilization gamma irradiation on the friction and wear properties of ultrahigh-molecular-weight polyethylene (UHMWPE) sliding against 316L stainless steel in dry air are summarized as follows:

1. The friction and wear properties of UHMWPE were not significantly changed by gamma irradiation doses of 2.5 and 5.0 megarads.

2. Characterization of UHMWPE by solution-viscosity techniques indicated that gamma irradiation increased the amount of insoluble gel (high-molecular-weight fraction) and also increased the amount of low-molecular-weight (<20 000) material.

3. After run-in, wear rate either was steady or gradually decreased as a function of sliding distance. In general, no late wear-rate increases were observed.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, January 15, 1979,

506-16.

APPENDIX - CHARACTERIZATION PARAMETERS

The following parameters were used to characterize the polymers in this study:

(1) Insoluble gel (high-molecular-weight fraction) - the percentage of the polymer that is not soluble in xylene at 135° C. This parameter represents crosslinked polymers and/or polymers with molecular weights of 6×10^6 or greater.

(2) Soluble fraction - the percentage of polymer that is soluble in xylene at 135° C. This parameter represents polymers with molecular weights of less than 6×10^6 .

(3) Low molecular fraction - the percentage of the total sample that has a weight-average molecular weight of about 20 000

(4) Intrinsic viscosity $[\eta]$ - the intrinsic viscosity of the soluble polymer in Decalin at 135° C

(5) Weight-average molecular weight \overline{M}_W - the weight-average molecular weight of the soluble polymer fraction according to the relation

$$[\eta]_{135^\circ \text{ C}} = 6.77 \times 10^{-4} \overline{M}_W^{0.67}$$

(6) Crystallinity (from density) - the percentage of the polymer that is crystalline, as determined by the gradient-column technique (ASTM D1505)

(7) Crystallinity (from DSC) - the percentage of the polymer that is crystalline, as determined by differential scanning calorimetry. Crystalline content was calculated for a heat of fusion of 284 joules per gram (68 cal/g) for polyethylene crystals

The following measurements were included in the characterization:

(1) Differential scanning calorimetry - A commercial DSC instrument was used at a heating rate of 10 degrees C per minute.

(2) Hardness measurements - Microhardness measurements were made on the wear-scar surface, at test conclusion, with a diamond pyramid indenter. The hardness number was calculated from the formula

$$\text{DPH} = 1.854 \frac{P}{d^2}$$

where P is 128 grams, d is the diagonal length of the square impression in millimeters, and DPH is the diamond pyramid hardness in kilograms per square millimeter.

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TABLE I. - SUMMARY OF TEST DATA

[Load, 1 kilogram (9.8 N).]

Radiation dose, megarads	Test	Total sliding distance, m	Surface velocity, m/sec	Average coefficient of friction	Average wear rate, $\text{m}^3/\text{N-m}$
0	1	40.0×10^3	0.061	0.55	2.5×10^{-15}
	2	22.2	.077	.51	2.2
	3	43.0	.073	.48	1.7
	4	45.0	.073	.34	.90
	5	45.6	.069	.45	.88
	6	182.0	.076	.40	.75
	^a 7	41.9	.074	.37	1.7
	8	130.0	.27	.47	1.7
	9	263.0	.26	(b)	1.5
	10	440.0	.27	.39	2.6
2.5	1	32.0×10^3	0.075	0.36	1.0×10^{-15}
	2	37.8	.064	.52	2.3
	3	42.2	.072	.42	1.3
	4	52.0	.074	.44	1.1
	5	185.0	.063	.45	.76
5.0	1	28.9×10^3	0.077	0.31	1.8×10^{-15}
	2	36.0	.070	.31	2.0
	3	36.7	.071	.41	1.6
	4	93.5	.076	.42	.78
	5	137.0	.076	.43	1.1
	6	133.0	.25	.33	1.1
	7	578.0	.24	.46	.63

^a2-Kilogram (19.6-N) load.

^bNot available.

TABLE II. - SUMMARY OF FRICTION AND WEAR TEST

DATA BY RADIATION LEVEL

Radiation dose, megarads	Number of tests	Average coefficient of friction	Average wear rate after run-in, $\text{m}^3/\text{N-m}$
0	10	0.44 ± 0.07	$1.6 \pm 0.66 \times 10^{-15}$
2.5	5	0.44 ± 0.06	1.3 ± 0.60
5.0	7	0.38 ± 0.06	1.3 ± 0.52

TABLE III. - CHARACTERIZATION OF POLYETHYLENE

TEST SPECIMENS

	Irradiation dose, megarads		
	0	2.5	5.0
Insoluble gel (high-molecular-weight fraction), percent	14	66	78
Soluble fraction, percent	86	34	22
Low-molecular-weight fraction, percent	1 to 2	4	5
Intrinsic viscosity (soluble), decaliters per gram	13.41	2.03	1.37
Weight-average molecular weight (soluble)	2 588 000	152 000	86 000
Density, kg/m^3	0.937	0.941	0.945
Crystallinity (from density), percent	68	70	72
Crystallinity (from differential scanning calorimetry), percent	46	49	50
Differential scanning calorimetry:			
Temperature at beginning of melt, $^{\circ}\text{C}$	101	101	101
Temperature at peak of melt, $^{\circ}\text{C}$	---	127 (minor) 133 (major)	125 (minor) 133 (major)
Temperature at end of melt, $^{\circ}\text{C}$	134	136	137
Diamond pyramid hardness, kg/mm^2	4.9 ± 0.7	5.2 ± 0.5	4.8 ± 0.5

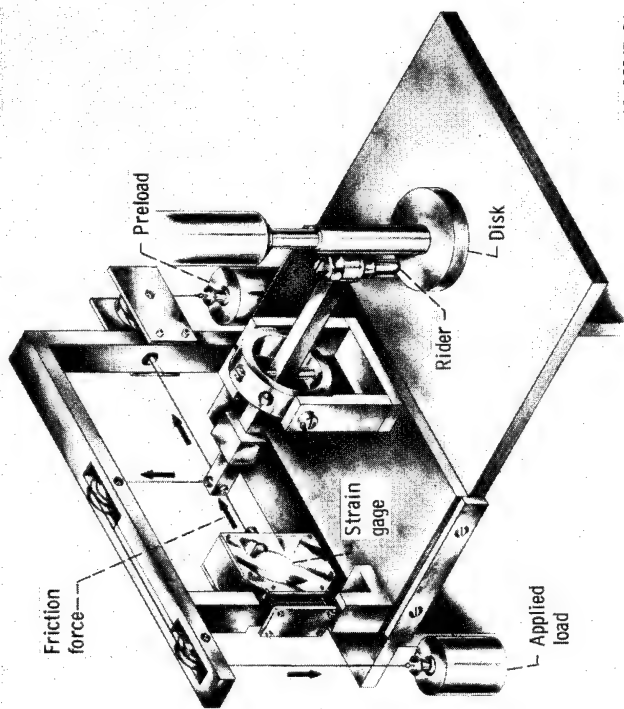


Figure 1. - Slow-speed friction rig.

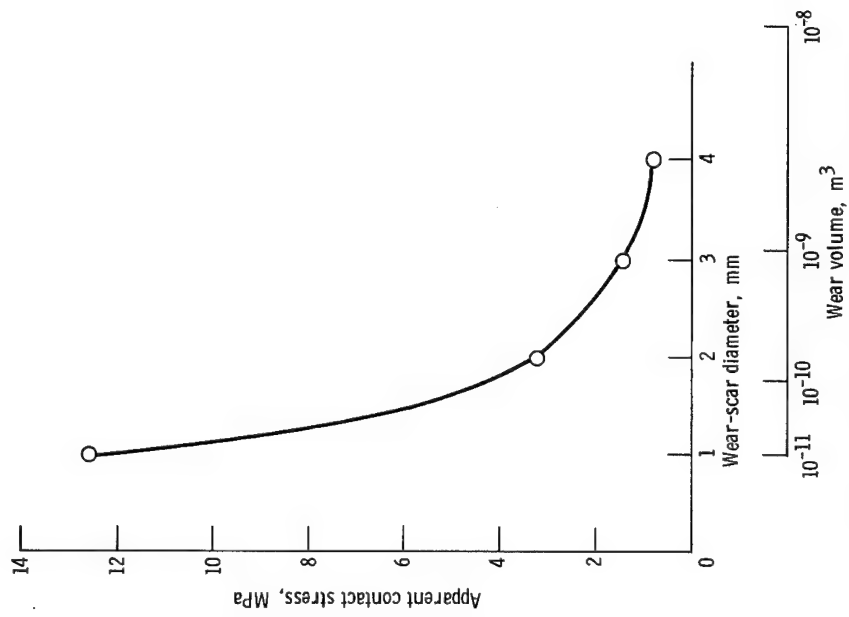


Figure 2. - Apparent contact stress as a function of wear-scar diameter. Load, 1 kilogram (9.8 N).

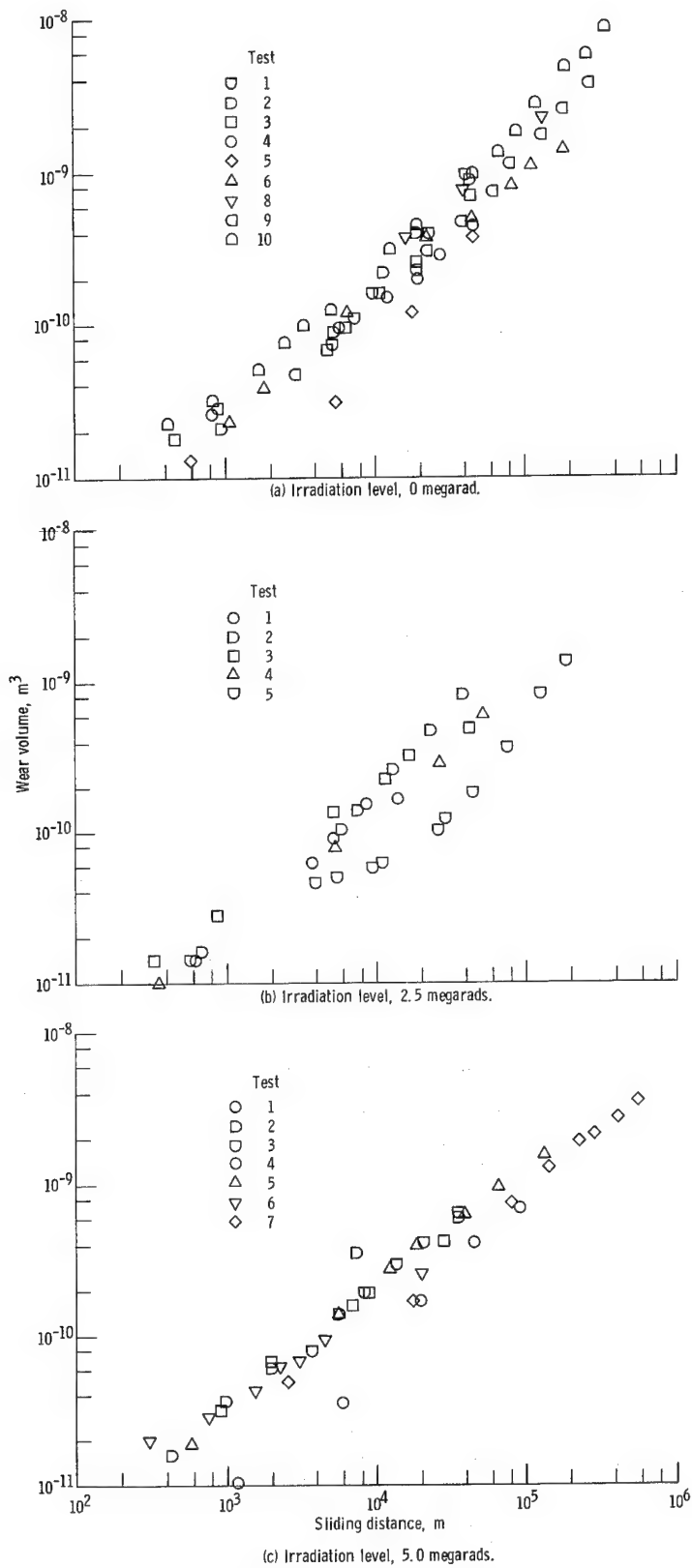


Figure 3. - Wear volume as a function of sliding distance.

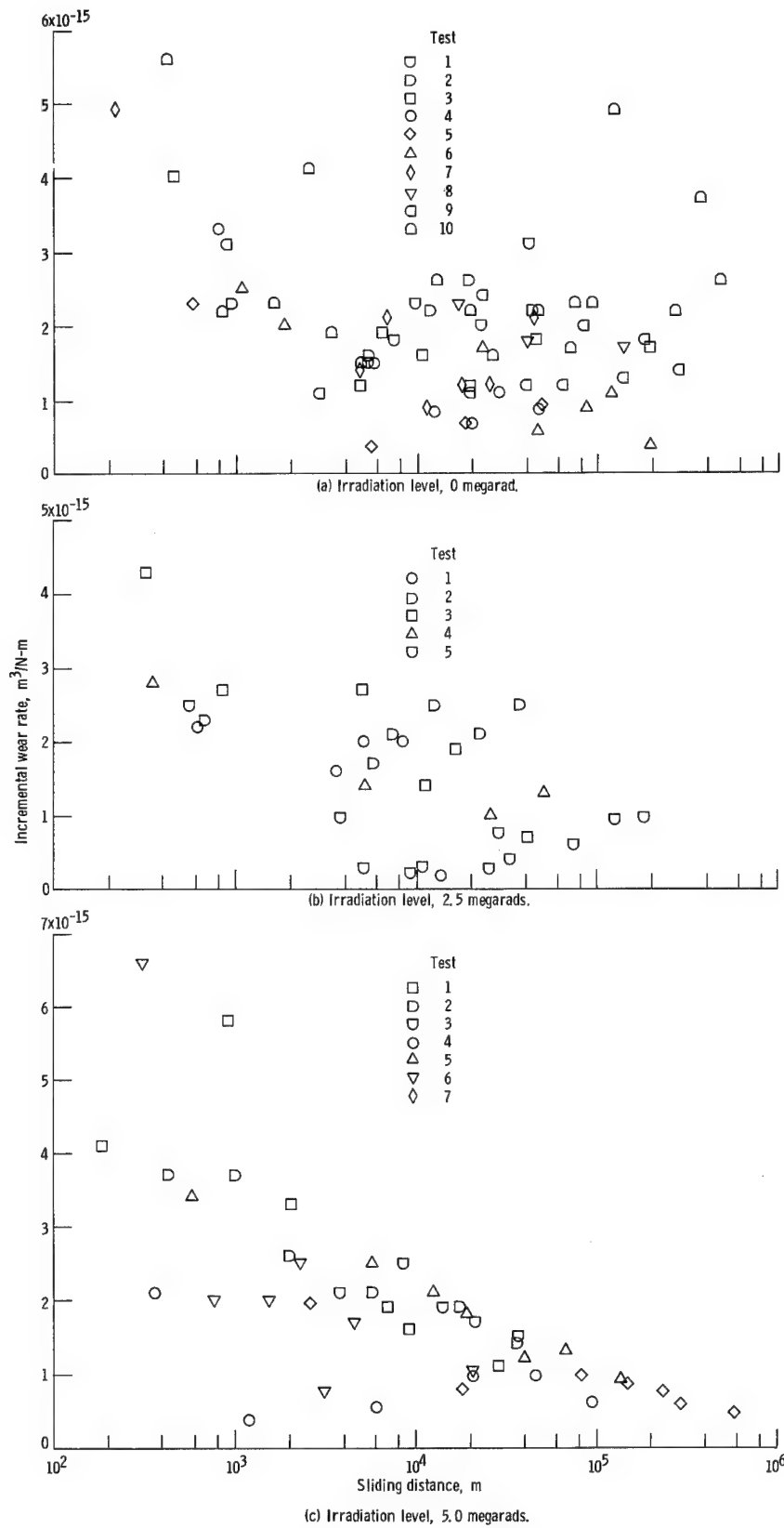


Figure 4. - Incremental wear rate as a function of sliding distance.

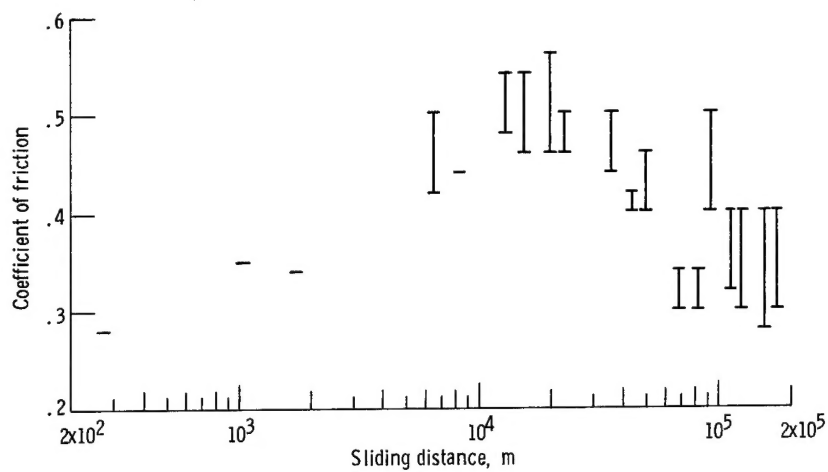


Figure 5. - Coefficient of friction as a function of sliding distance for test 6 (0 megarad). Load, 1 kilogram (9.8 N); sliding velocity, 0.076 meter per second.

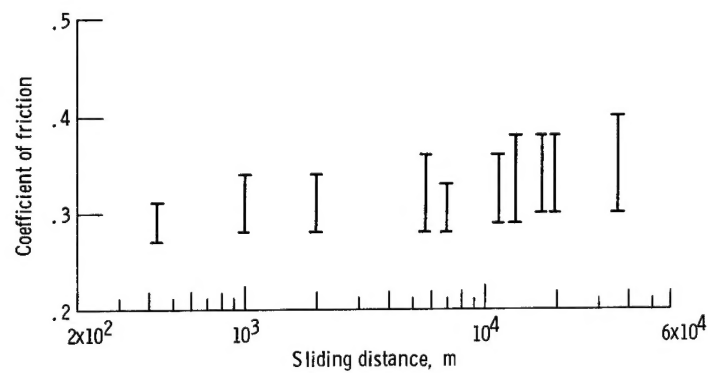


Figure 6. - Coefficient of friction as a function of sliding distance for test 2 (5.0 megarads). Load, 1 kilogram (9.8 N); sliding velocity, 0.070 meter per second.

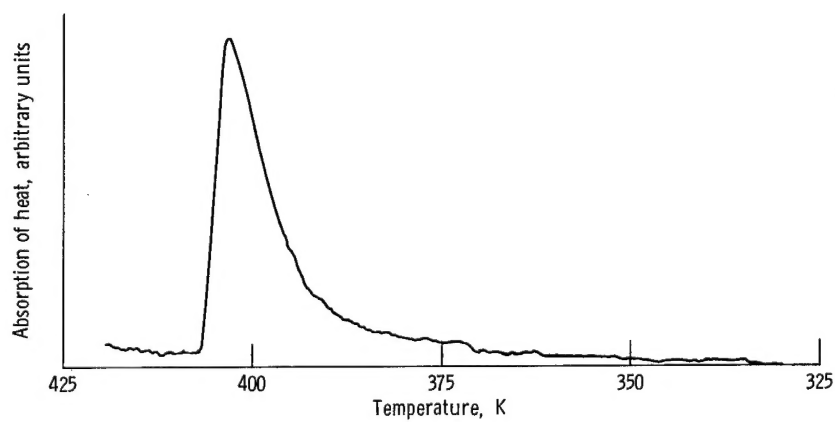


Figure 7. - Differential scanning calorimetry curve for unirradiated polyethylene.

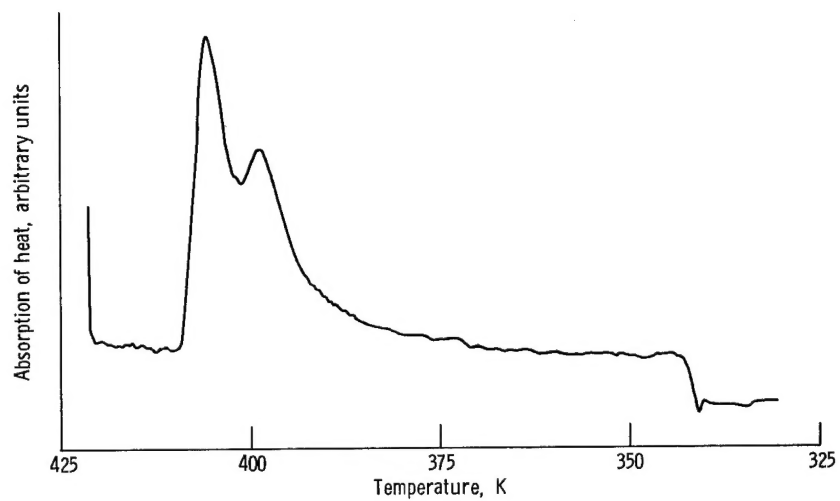


Figure 8. - Differential scanning calorimetry curve for irradiated polyethylene (2.5 mega-rads).

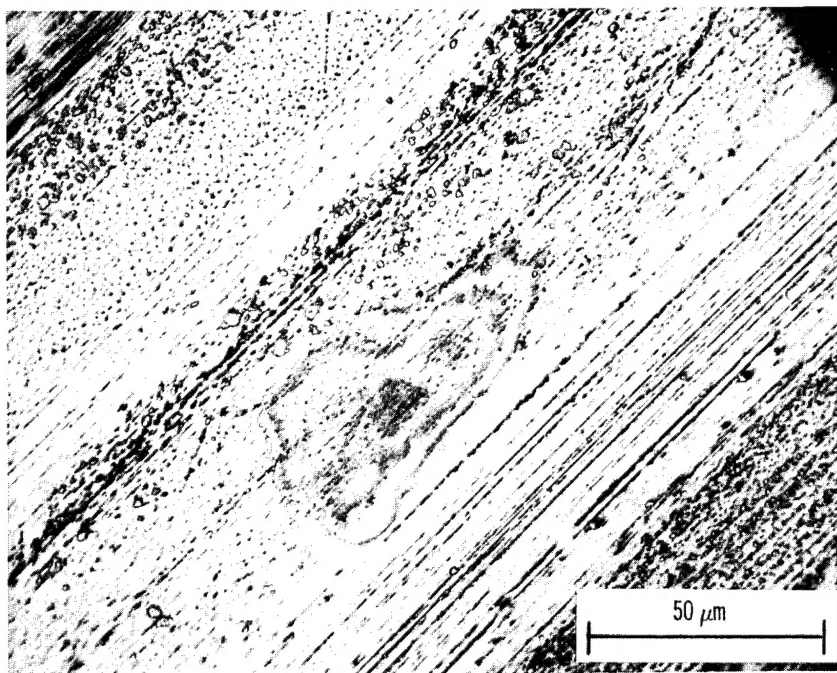


Figure 9. - Transfer film on 316L steel disk after 2500 meters of sliding.

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16. Abstract The effect of sterilization gamma irradiation on the friction and wear properties of ultrahigh-molecular-weight polyethylene (UHMWPE) sliding against 316L stainless steel in dry air at 23° C was determined. A pin-on-disk apparatus was used. Experimental conditions included a 1-kilogram load, a 0.061- to 0.27-meter-per-second sliding velocity, and a 32 000- to 578 000-meter sliding distance. Although sterilization doses of 2.5 and 5.0 megarads greatly altered the average molecular weight and the molecular weight distribution, the friction and wear properties of the polymer were not significantly changed.					
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